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**The Effects of Potato, Corn and Wheat Starch on the Rheological Properties
of Pigmented Paper Coatings**

by

Brian G.Hart

**This Thesis is submitted
in partial fulfillment of
the course requirements for
The Bachelor of Science Degree in Paper Engineering
Department of Paper and Printing Science and Engineering**

**Western Michigan University
Kalamazoo, Michigan
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ABSTRACT

The paper industry is one of the largest users of commercial starch in the world. Starch has many uses in the papermaking process, including coating operations. In coating, starch has two main purposes: to work as a binder and to give desired rheological properties to the pigmented paper coating. Research was done to examine the effects of three different starches on the rheological properties of pigmented paper coatings. The three starches were corn, wheat, and potato. These starches were modified using the enzyme conversion process. The converted starches were added to the coating formulas and then tested for high and low shear rate viscosity. The Hercules and Brookfield Viscometers were used to measure the corresponding viscosities and thixotropy. The evaluation showed that the potato starch had the greatest effect on the viscosity and thixotropy of the coatings, followed by the wheat and the corn.

Keywords: Potato Starch, Wheat Starch, Corn Starch, Coating Latex, Viscosity, Rheology.

Introduction

Starches have a wide range of use in the paper industry. Starches are used widely in size press and publication coatings in the United States because of economic considerations and because of the acceptable properties they contribute to the coating. Starches in coatings generally have two functions: to work as a binder and to assist in the flow properties of the coating.

Coating binders fall into three categories: Starches, proteins, and synthetics. Starches continue to be replaced by synthetics which are mainly latexes (based on styrene-butadiene, acrylic or vinyl acetate polymers). Protein binders are either casein (isolated from acidified skim milk), soy bean extracts, or in rare cases animal glues. Additives are employed to meet specific requirements of the coating equipment and product end use.

In this Research starch was the only binder investigated. A comparison was made of the rheology that different starches give to the coating formula. These starches were: wheat, corn, and potato.

Starches: A General Discussion

Starch is a carbohydrate synthesized in corn, wheat, potato and other plants by the polymerization of dextrose units (1). This polymer exists in two forms: A linear structure of about 500 units called amylose and branched structure of several thousand units called amylopectin. These structures are given in figs. 1a and 1b. The amylose is a D-glucose polymer bounded by 1-4 alphasugcosidic linkages, and amylopectin contains mostly 1-4, alphasugcosidic with alpha 1-6 linkages at the branch points. The differences in enzyme conversions of the different starches as well as the rheology are caused by these properties.

Starch is supplied as a white, (or in the case of corn, yellow) granular powder which is insoluble in cold water because of the polymeric structure and hydrogen bonding between adjacent chains. However, when the aqueous dispersion is heated, the water is able to penetrate the granules and cause them to swell, producing a “gelatinized” solution or paste (2). This swelling causes the granules to increase in size by about 50% or more depending on the type of starch. The gelatinization of the starch is required to properly convert the starch using enzymes, for starch application at the size press, and for coating applications. Cooking can either be done through batch or continuous operations.

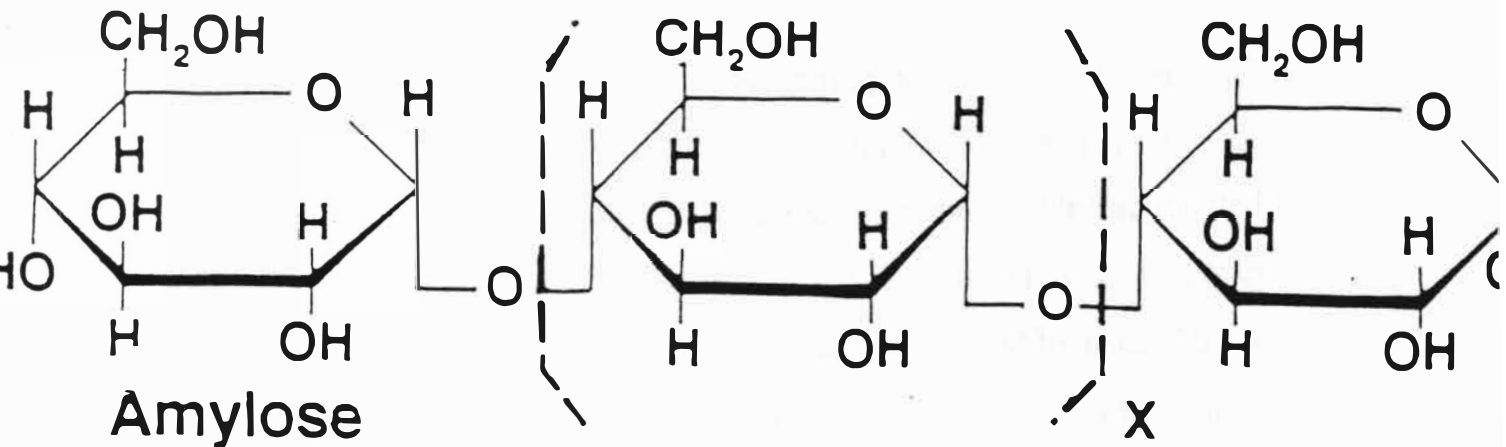


Figure 1a - Chemical structure for amylose (2)

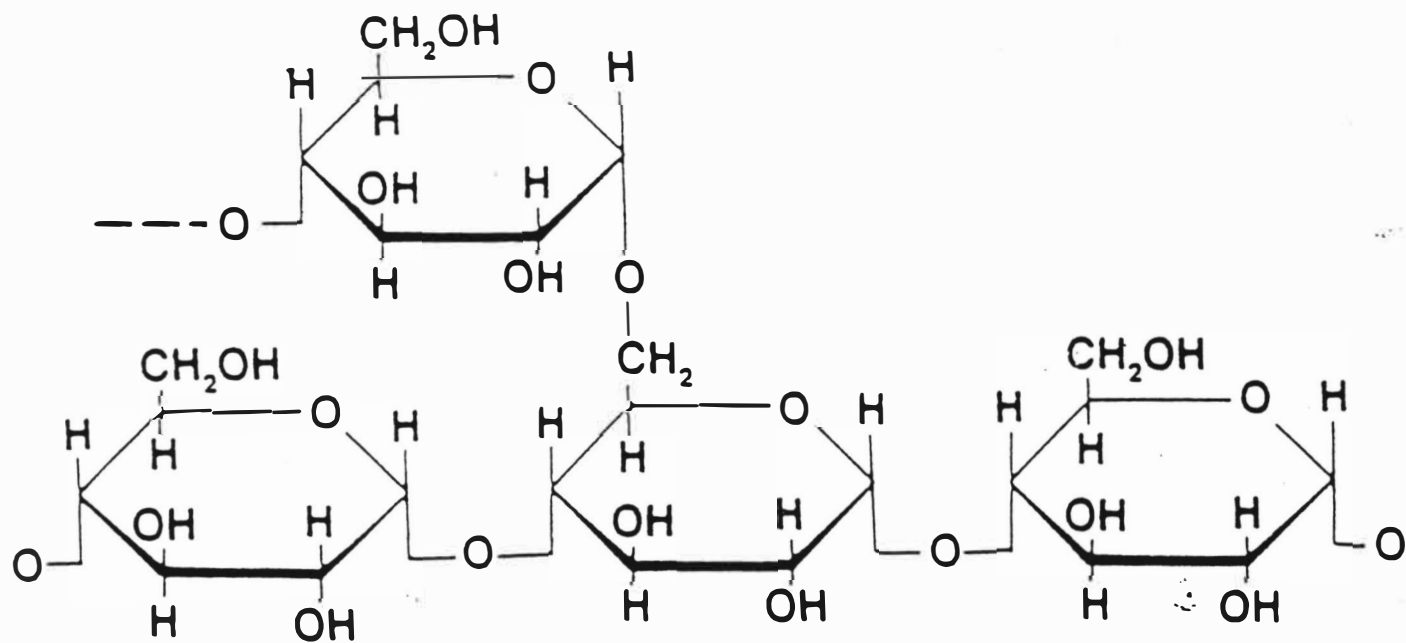


Figure 1b - Chemical structure for amylopectin (2)

Cooling the hot solution causes thickening which is called “setback” or “retrogradation”. To prevent setback it is recommended that these starches be stored at temperatures of about 150 degrees Fahrenheit. Unfractionated and unmodified starch, called “pearl starch”, is viscous and has a tendency toward setback, even without cooling. Set-back is avoided by using 100% amylopectin, “waxy” starch which forms a clearer paste and is non-gelling; however, a loss of sizing efficiency results because the linear fraction contributes more toward film formation.

There are several conversion methods that can be applied to unmodified starch to give the solutions lower viscosity and resistance to retrogradation. These processes included chemically oxidizing the starch, adding hydroxyethylether groups to the starch, or enzyme converting the starch. The enzyme conversion process can create a low viscosity starch with film forming properties and retrogradation resistance maintained at a reasonable level.

Corn Starch

In recent years the United States has produced starches at a rate of over four billion pounds per year. Corn starches account for over 90% of this total. The paper industry uses over 50% of the starch consumed in the United States, with food and textile being the other major consumers.

Corn kernels are made up of three main parts, the pericarp, the starchy endosperm, and the embryo (or the germ). The pericarp is the outer skin or hull of the kernel which serves to protect the seed. The endosperm, the main energy reserve of the corn kernel, makes up about 80% of the total weight of the kernel. In the endosperm 90% is starch and 7% gluten or protein, with a small amount of oil and other constituents. Corn starch is classified as a cereal seed starch. The component granules are round polygonal in shape (3).

Figure 2 is a schematic outline of the wet milling process for corn. The first step in the isolation of starch from corn by this “wet Milling” process is “steeping” which means to swell or soften the kernel so that the hulls, fiber, germ, gluten, and starch can be separated. A course grinding operation after some screening loosens the hulls and frees the germs, which contain almost all the oil. Since the starch granules settle more rapidly than the gluten they are then separated from each other and concentrated by a series of screening and centrifuging operations (3).

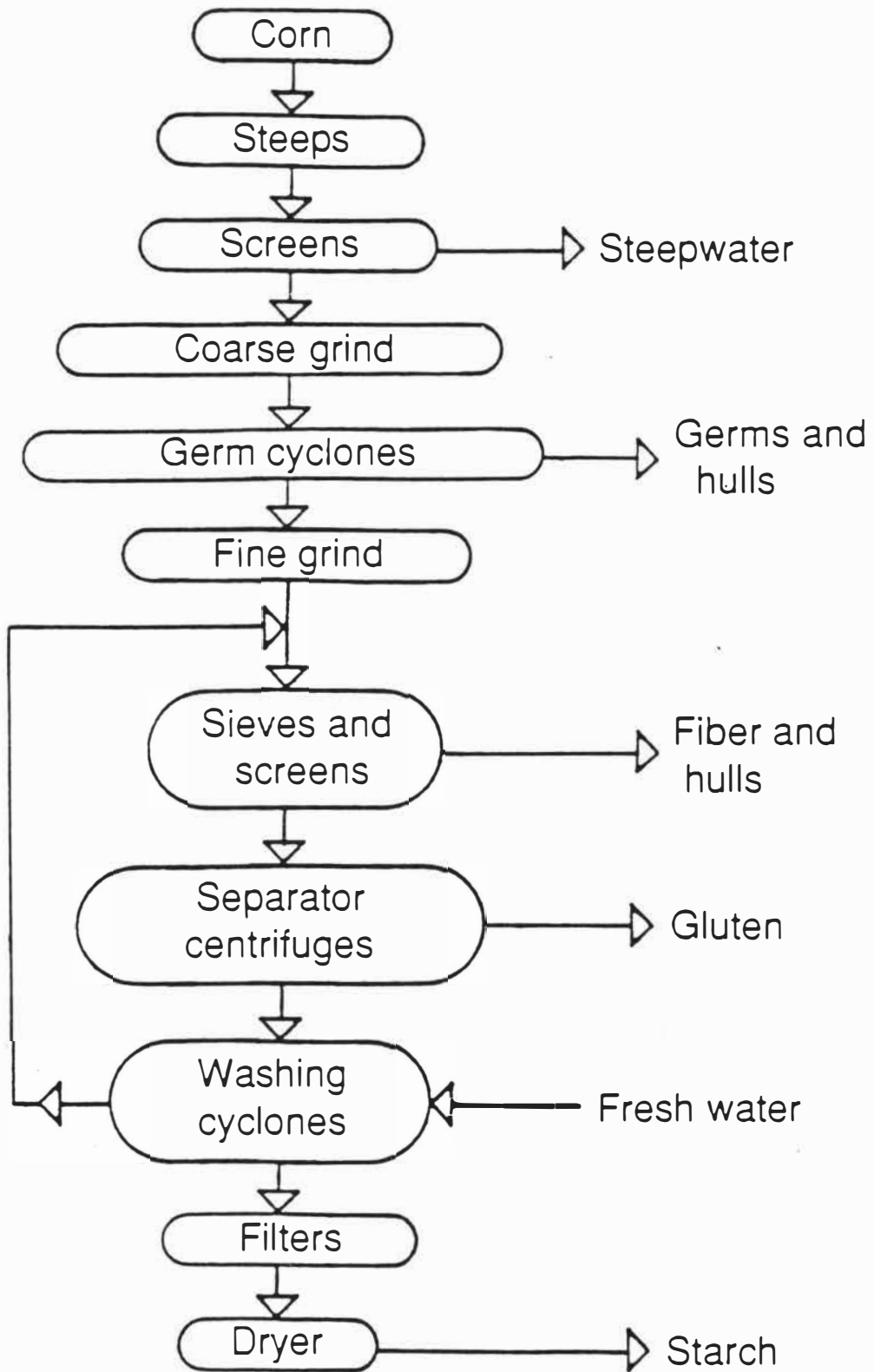


Figure 2 - Wet milling process for corn (2)

The final separation is done through a series of washing cyclones. The granules are then vacuum filtered and dried by belt or spray dryers. If the starch produced by the milling process is simply dried, it is called “pearl” or unmodified starch.

There are certain physical and chemical properties that distinguish starches from one another. For the chemical properties of corn starch, the amount of amylopectin is about 72%. The degree of polymerization in corn starch is 8000 for amylopectin. The physical composition of corn starch is 17% moisture, 60% starch, 8% protein, 2% fiber, 4% fat, And 1.2% other materials. Commercial isolated starches contain about 12% moisture and about 99% starch (on a dry basis).

Once the starch is heated and forms a paste there are certain properties associated with these pastes. The corn starch starts to gel at about 170 degrees Fahrenheit. Corn starch forms a paste of medium viscosity which has a yellowish, opaque look. Its rate of retrogradation is high and the resistance of molecules and networks to shear degradation is medium in comparison to other starches.

Wheat Starch

In most countries wheat is the dominant crop of choice, Therefore, wheat plays an important role as a raw material for the production of starch. In the United States corn is the major source of starch. Both are categorized as a cereal starches and both exhibit similar chemical and physical properties. The manufacture of wheat starch is more economical than corn because gluten, which is by product of wheat starch can be recovered (4).

In the manufacturing process to obtain wheat starch, wheat flour is the raw material. This process involves the addition of water to the flour to create a dough ball at a consistency of about 40%. The dough ball is then rolled, kneaded and sprayed with additional water. The washing step takes the starch away from the gluten. The starch is then refined and dried (4).

The amylose and amylopectin content of wheat starch are the same as corn at 28% and 72% respectively. The degree of polymerization of amylopectin is also at 8000. The average granule size is slightly less than corn at 10 micrometers.

Wheat starch requires a gelatinization temperature of about 165 degrees Fahrenheit. This is slightly lower than corn, but higher than at 140 degrees Fahrenheit. The wheat starch paste has a medium-low viscosity. The appearance of the paste is cloudy and white. Its resistance to shear is medium (6).

Potato Starch

America was the leading producer of potato starch in the nineteenth century, but production has fallen off in recent years. Potato starch has many advantages and is widely produced in this country.

The techniques and equipment used in the isolation of potato starch vary widely from one processing plant to another. A typical process for extracting potato starch from potatoes is as follows.

In the manufacturing process the potatoes are first thoroughly washed to remove the dirt and sand. Then they are disintegrated by rasping or grinding to liberate the starch granules from the skin and fiber. Sulfur dioxide is added to the ground mixture to inhibit enzyme and bacterial activity as well as discoloration of the starch. The fiber and skin are removed in a series of screening or sieving steps. Continuous centrifuges are used to remove protein and soluble materials. The starch is dewatered with rotary vacuum filters and the filter cake is dried in flash or cyclone dryers.

Potato starch is classified as a tuber starch. The component granules are oval in shape. The amylose content in the starch is about 21% and the amylopectin content is about 79%. The degree of polymerization of amylopectin is about 30,000 which is almost three times more than that of

corn and wheat. The average particle size of the potato granule is 30 micrometers.

When the starch solution is heated the gelatinization temperature of about 140 degrees Fahrenheit is lower than that of wheat and potato. The paste viscosity is very high and has a clear appearance. Its resistance to shear is medium-low and rate of retrogradation. Also the presence as small amounts of phosphate groups leads ionic stability to the dissolved molecules.

Enzymes

The use of enzymes for Industrial Processes goes back to brewing of beer as early as 500 B.C.. In the industrial world of today enzymes are used in many kinds of processes and applications. This research concentrates on the use of enzymes for starch in paper coatings.

Enzymes constitute a very important group of catalyst or chemical activators, which have great capacity for influencing chemical change without actually entering the chemical reaction. An enzyme is a biocatalyst. A biocatalyst is an organic substance produced by living matter (in this case, bacteria or molds) which has the ability to accelerate a specific chemical change without itself being affected.

The type of enzyme used for this research is a hydrolytic enzyme which acts upon starches. There are two general classes of starch converting enzymes: alpha amylases (liquefying enzymes) and beta amylases (saccharifying enzymes) (8). For this research the first mentioned class will be used. This acts on the alpha 1-4 bonds in the starch polymers. The converting strength of a starch liquefying enzyme may be expressed in terms of a unit known as the "liquefon". A liquefon is defined as that quantity of enzyme which will liquefy a special starch substrate under specific set of reaction conditions at an initial rate of 25mg. of starch per minute (9). The enzyme used for this project was supplied at a standard strength of 20,000 liquefons per gram.

Rheology: A General Discussion

“Rheology is the study of the deformation of flow of matter. In the study of the behavior of materials there are two qualitatively different kinds of measurements. The viscometric experiments, which measure a single material constant - the viscosity - usually as a ratio of shear stress to the shear rate, and, the rheometric measurements, where rheologists develop a number of carefully controlled experiments to identify one or more components of stress tension in well defined flows of materials that do not obey the Hookean law of elasticity or the Newtonian law of viscosity¹”.

“Rheology is important in preparation and assessment of paper coating “colors”, a blend of mineral pigments, binders, and polymeric additives, since experience has shown that control of the color viscosity to within certain limits results in a desired coat weight and satisfactory coating layer. Processes involved in paper coating operations are preparation, transportation, and application of color, where the ultimate goal is to continuously apply a uniform layer of liquid with well-controlled thickness to the paper. Although similar processes are involved in coating substrates in film form, paper is one of the most complex systems due to its compressibility and porosity.²

¹ Nicolas Triantifilopoulos, “shear Induced Anisotropy in the Rheology of Solids Dispersions” (Maser Thesis, Western Michigan University), 25-32.

² (Triantifilopoulos 1985, 25-32)

Viscometers: Hercules High Shear and Brookfield

The Hercules Hi- Shear viscometer (HHSV) utilizes concentric cylinders with a well defined geometry to measure a fluids resistance to flow and determine its behavior in this simple-shear flow field (fig 3) (11). Because the gap between the rotating inner bob and the restrained outer cup cylinders approximates a velocity-driven Couette flow. This type of flow is similar to the one confined between two parallel plates, where the one moves relative to the other. When a fluid sample is confined between the bob and the cup, rotation of the bob generates a velocity gradient across the gap. This gradient is termed shear rate and is defined as the change in linear velocity between two fluid elements divided by there distance in centimeters. Thus, shear rates are expressed in reciprocal seconds. While rotation of the bob causes the fluid to flow, its resistance to deformation imposes a shear stress on the inner wall of the cup, measured in dynes/cm².

The Brookfield Viscometer is of the rotational variety (12). It measures the torque required to rotate an immersed element (the spindle) in the fluid (fig 4). The spindle is driven by a synchronous motor through a calibrated spring; the deflection of the spring is indicated by a pointer and dial. By utilizing a multiple speed transmission and interchangeable spindles, a variety of viscosity ranges can be measured, enhancing the versatility of the instrument.

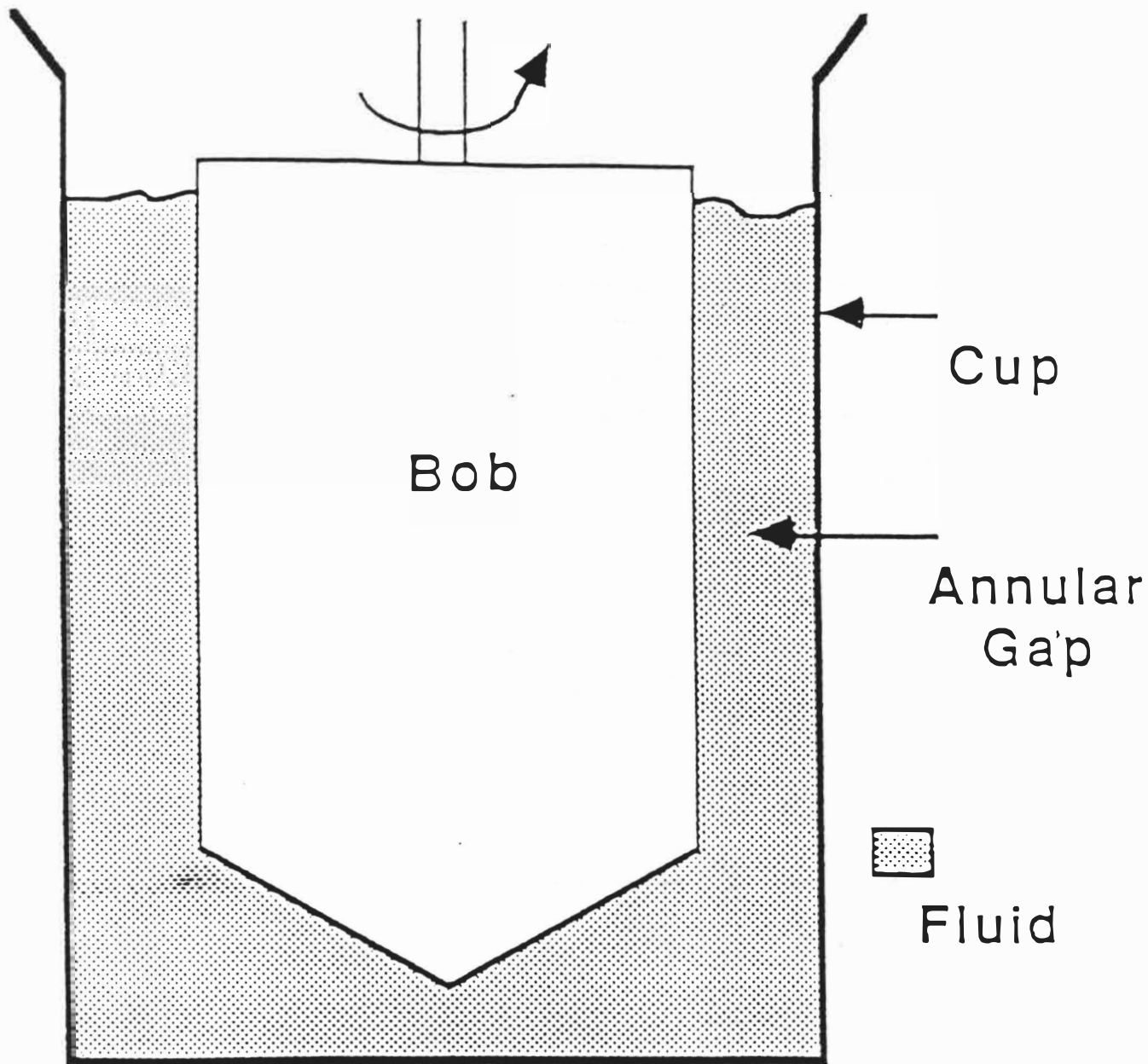


Figure 3 A bob and cup configuration for HHSV (11)

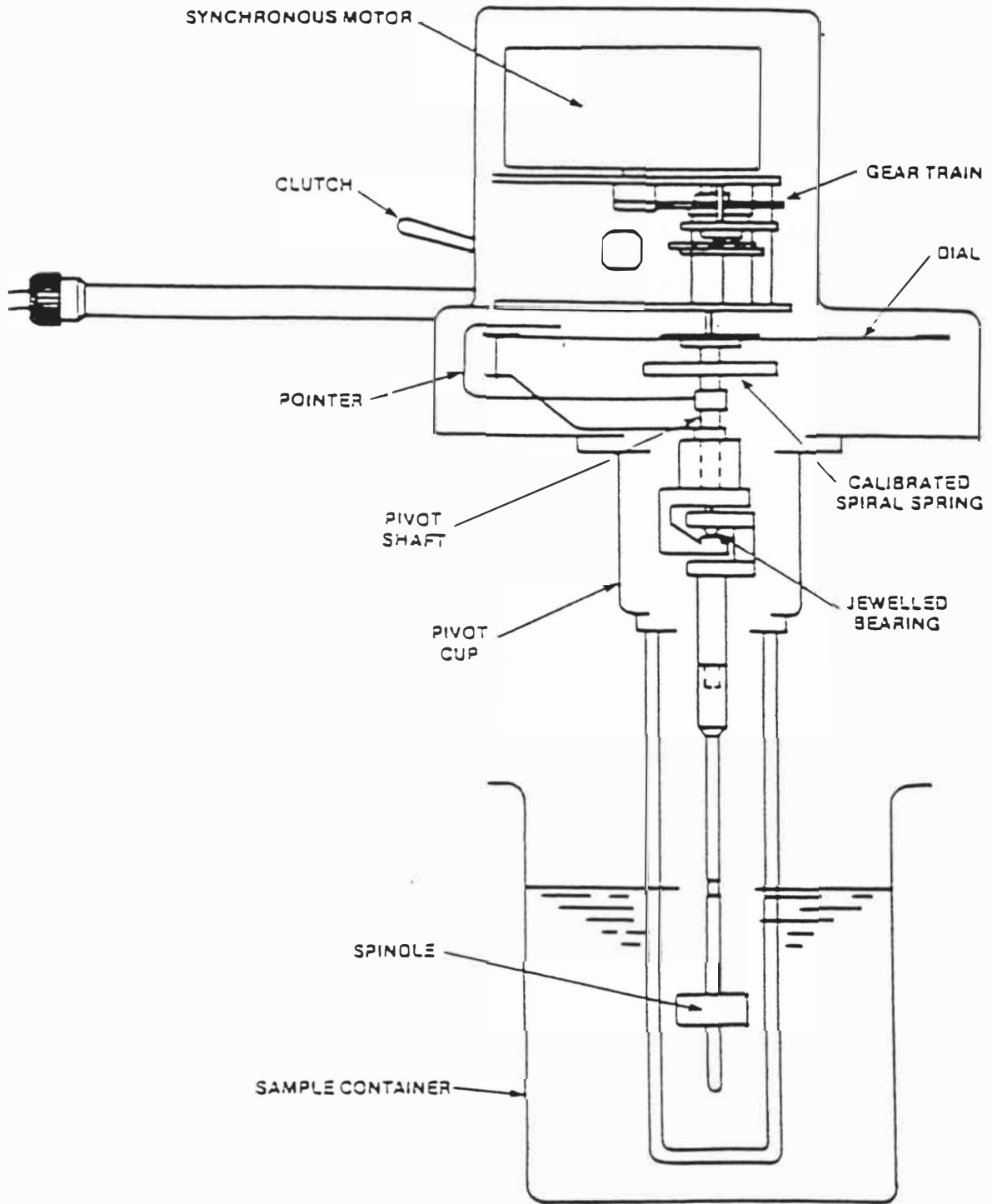


Figure 4 Brookfield Viscometer (12)

For a given viscosity, the viscous drag, or resistance to flow is proportional to the spindle's speed of rotation and is related to the spindle's size and shape (geometry). The drag will increase as the spindle's size and rotational speed increases. It follows that for a given spindle geometry and speed increases. It follows that for a given spindle geometry and speed, an increase in viscosity will be indicated by the increase in the deflection of the spring.

Problem Statement

Starches are used in paper coating to give a desired flow property and as a binder. For many years starches have been examined as a certain group of binders with some experimentation done in the way of their individual flow characteristics on pigmented paper coatings. The three starches examined (corn, wheat, and potato) are commonly used starches in the paper industry. Testing was done on these three starches to determine their rheological properties in pigmented paper coatings.

Experimental Design

Design

This is an outline of the experimental work that was done:

- A. Conversion of the corn, wheat and potato starches using the enzyme conversion process.
 - 1. Testing the viscosity of the enzyme converted starches on the Brookfield and Hercules High Shear viscometer.
- B. Preparation of two different coatings for testing.
 - 1. Using starch as the only binder (20 parts dry starch per 100 parts pigment).
 - 2. Using a 50-50 starch latex combination (total 20 parts)
- C. Testing the coatings for rheology.
 - 1. Test on the Brookfield viscometer.
 - 2. Test on the Hercules High Shear Viscometer.

Materials

This is an outline of the materials used for the experiment

- A. Starches used (all modified)
 - 1. Corn
 - 2. Wheat
 - 3. Potato
- B. Vanzyme of R.T. Vanderbilt Co.
- C. Dow 620 Latex
- D. Hydrasperse, a #2 Clay, Huber Co.
- E. Nopcote C-104 (calcium stearate)

Procedures

The main procedure for this experiment required the process of converting the unmodified starch with enzymes. The enzyme used was Vanzyme which is a hydrolytic enzyme.

The first part of the procedure was to create a starch solution with 30% solids content. The Vanzyme (which was in powder form) was prepared as a 1% solids aqueous solution. The addition rate of the enzyme to the solution was 0.05% (based on dry starch), which was based on preliminary experimentation to ensure for proper conversion. This conversion process was done at 30% solids. The pH of the dispersion was checked and adjusted to the range of 7 to 7.3. If the solution was below this range soda ash was added to bring it up to the proper range. The time temperature cycle then was employed for proper conversion. This cycle was a three step process similar to that in fig. 4.

In the first part of this cycle the starch-enzyme solution was heated to an appropriate cooking temperature. This temperature was 170 to 185 degrees Fahrenheit for the wheat and corn starches and 160 to 165 degrees Fahrenheit for the potato starches. The solution then gelled and was held for five minutes. The temperature increased a few degrees during the holding period.

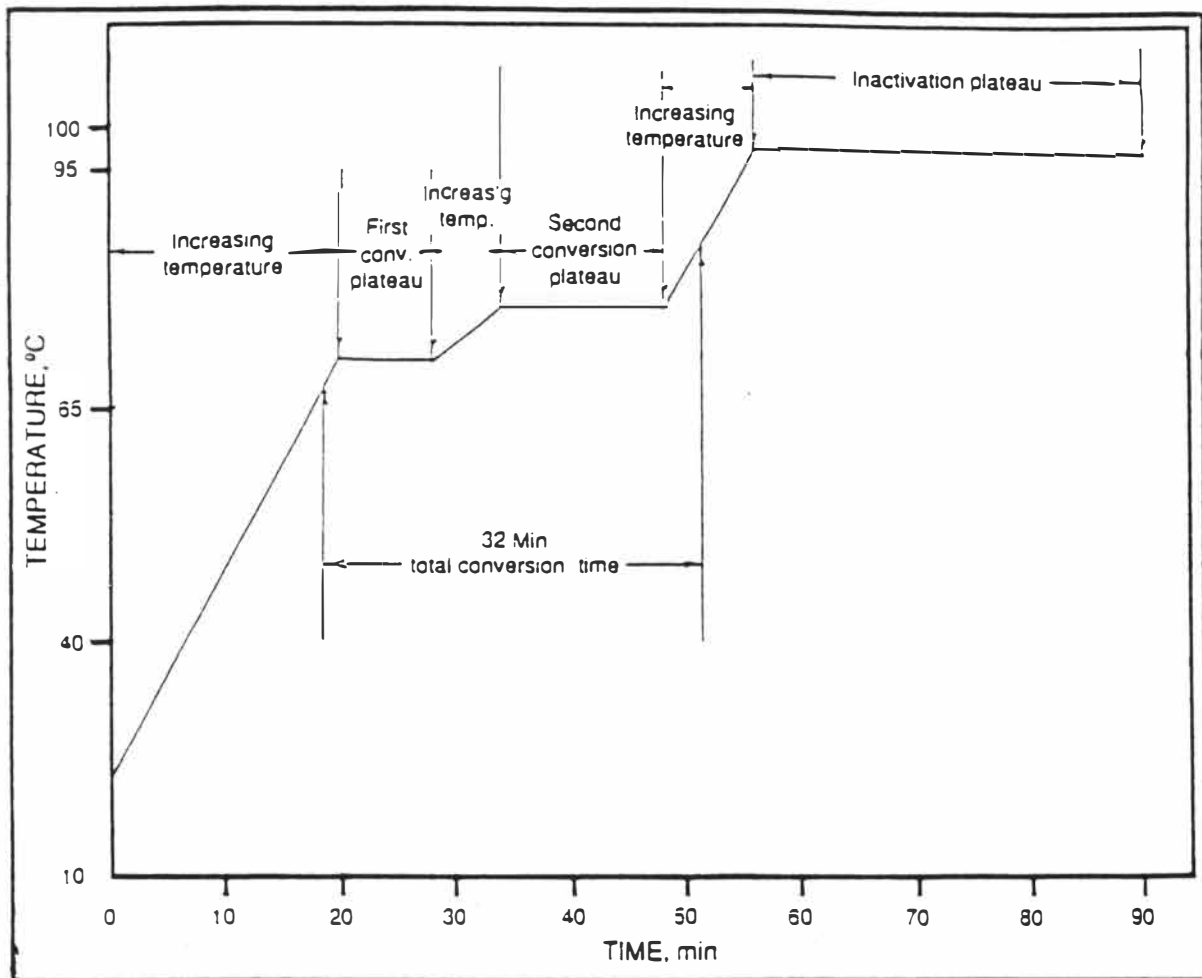


Figure 4 - A typical cooking curve for enzyme converted starches (2)

There are two different stages for the conversion process with a total conversion time of 30 minutes. The solids were then adjusted to 25% and the temperature controlled to 150 degrees Fahrenheit. The Brookfield was then checked on the solution to obtain the proper viscosity. The temperature was then raised to 200 degrees and held for ten minutes to inactivate the enzyme. This completed the conversion process.

The next procedure was the preparation of the coatings.

All the coatings were made under two basic formulations, one using starch as the only binder, and the other being a 50-50 combination of starch and latex. Table 1 and 2 show the actual coating composition of the two formulas. The coatings were adjusted to 54.8% solids by adding water.

The last procedure includes testing of the converted starches on the Brookfield and Hercules Hi-shear for viscosities, as well as determining the rheograms. These samples were tested at 150 degrees Fahrenheit. For the Brookfield viscometer the proper spindle was selected as well as the proper speed (100 rpm). The starch solution was then placed in a container and the spindle was immersed. The instrument then gave the viscosity readings in cp. units. The readings were kept in the 40-60% range of the scale. The starches were enzyme converted to a target of about 350 cp. on this viscometer.

Table 1
Coating Formula with Starch as only Binder

Material	Parts	Dry Wt.	%Solids	Total Wt.
#2 Clay	100	250g	72	347g
Starch	20	50g	25	200g
Nocote	0.5	1.25g	40	3.1g
Total		301.25g		550.1g

Table 2
Coating Formula with 50/50 Starch-Latex Combination

Material	Parts	Dry Wt.	%Solids	Total Wt.
#2 Clay	100	250g	63	396.75g
Starch	10	25g	25	100g
Latex	10	25g	50	50g
Nopcote	0.5	1.25g	40	3.1g
Total		301.25g		549.9g

units. The readings were kept in the 40-60% range of the scale. The starches were enzyme converted to a target of about 350 cp. on this viscometer.

After the starches were properly converted to the target range they were then subjected to the Hercules viscometer. The first set of starches were run without any pigment or latex binder added to the solution. The parameters at which the bob rotated were preselected to reach a maximum 4400 rpm's in a 21 second period. The return cycle also took 21 seconds. The computer in the system collected all the data points for each cycle interval. These data points were then used by the on board computer to construct the rheograms. All these starch pastes were converted to have similar Brookfield readings of 350 cps prior to being subjected to the Hercules.

The second and third set set of rheograms were run on the final coatings for each starch, coating pigment, latex, and calcium stearate.

Discussion of Results

The purpose of this study was to determine the effects of different starches on the rheological behavior of coating colors. The measurements taken were from viscometers. The objective of measuring the viscosity of the fluids was to construct rheograms to study the flow behavior of the materials. These rheograms can supply direct information regarding yield stress shearing history and thixotropy. In this study the material examined was studied to show the effects that different starches had on the viscosity and thixotropic properties of the material.

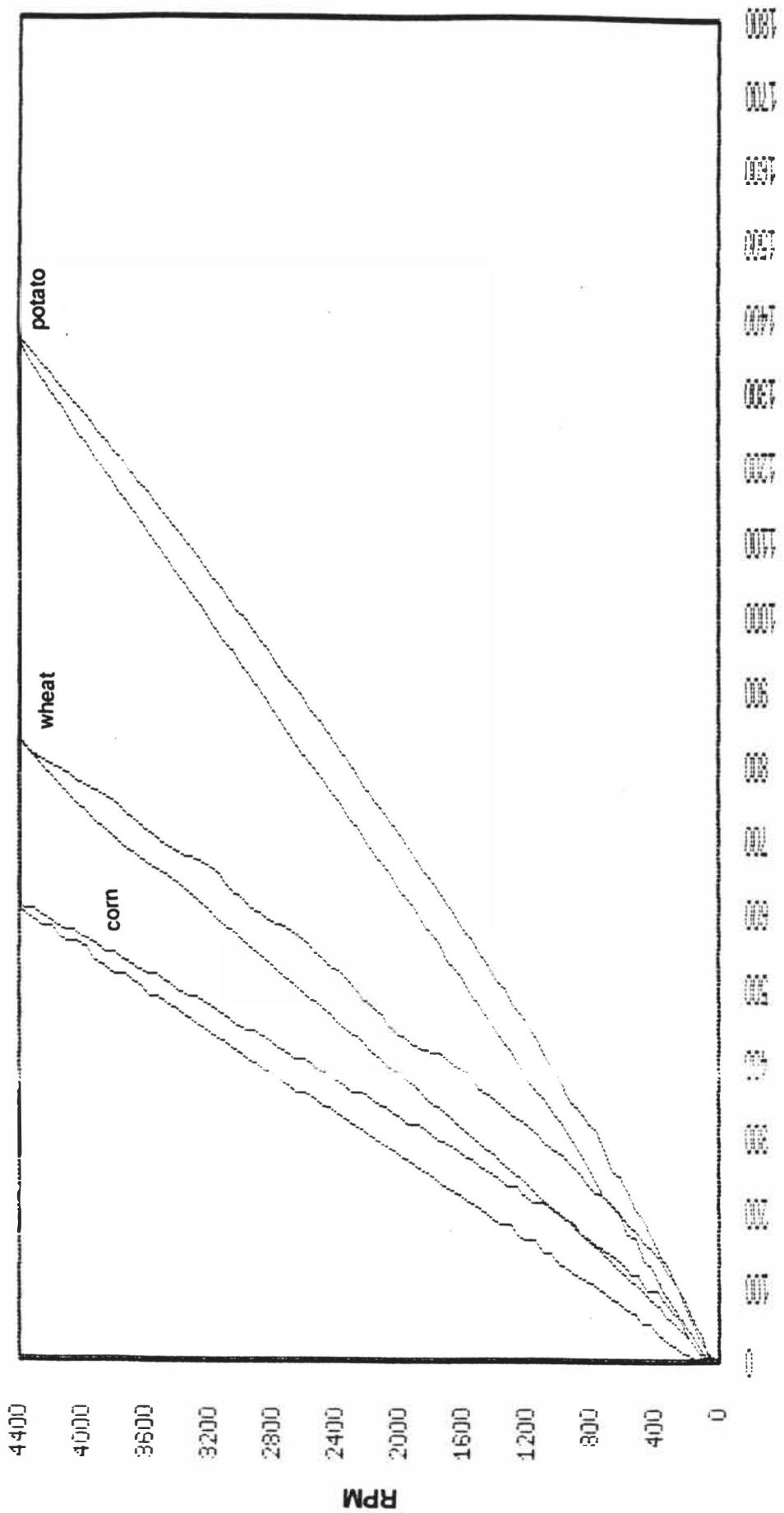
Thixotropy is the phenomenon where the increasing and decreasing shear rate flow curves do not coincide (Fig. 5). The development of the hysteresis loop depends on previous shear history and both the rate of change and the maximum shear rate. Many materials are thixotropic at high shear rates.

There were two rheograms constructed for each of the three sets of runs. The first set was the starch pastes, the second was the coating with the starch as the only binder, and the third was starch and latex as the binders. The first plot (Fig 6) is shear rate vs. torque and (Fig 7), is viscosity vs. shear rate. Figure 6 shows the thixotropic properties of the material where the curve furthest to the right with the up arrow indicating the shear rate as the rpm's increase. The down curved occurred after the

maximum rpm's was reached. Figure 7 shows the viscosity as a function of shear rate after the up and down curves of the first plot with the highest line going to the right representing the viscosity as a function of shear rate and the down curve representing the viscosity down trend as a function of shear rate. For the starch pastes alone (Figs. 6 & 7) the potato starch gave the highest values for the amount of high shear viscosity and the coefficient of the thixotropic breakdown for all the starches. This was followed by wheat and then corn.

Once the starches were added to the coating formulas their viscosity values increased (Figs. 8 & 9). The effects that the different starches had on the coatings followed the same ranking as the pastes themselves, except the viscosities were much higher. The potato starch gave the highest change in the coating by a large margin. The amount of conversion done to the potato starch was increased by, increasing the amount of enzyme used, so that all the coatings had similar Brookfield readings.

EPO11.DAT
E01HEAT.DAT
E00R011.DAT



TORQUE Kilodyne-cm

Figure 6 - RPM vs. Torque on Starch

ECORN1.DAT
EMWHEAT.DAT
EPOT1.DAT

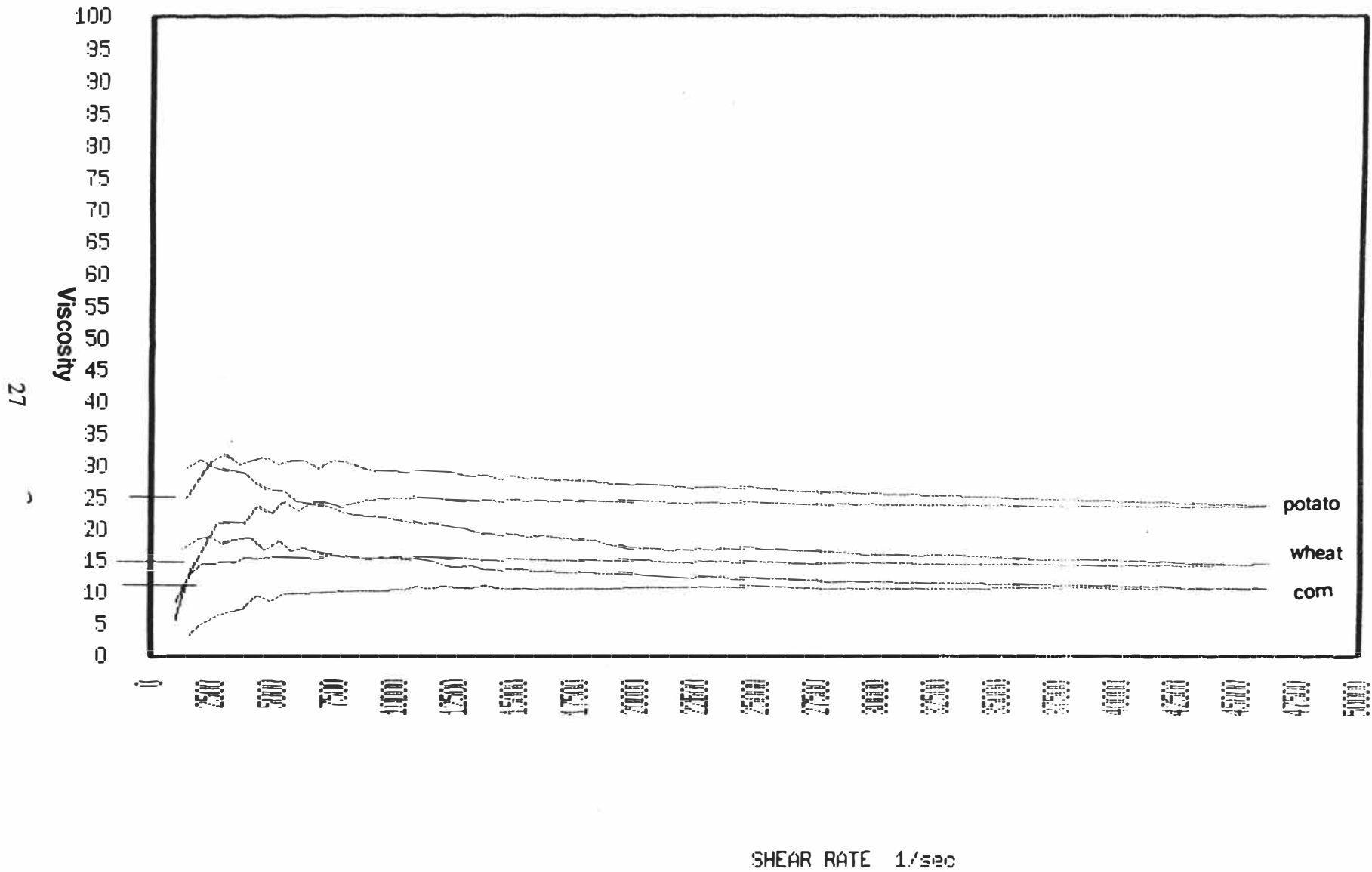


Figure 7 - Viscosity vs. Shear Rate on Starch

EPOT2.DAT
EWHEAT2.DAT
ECORN2.DAT

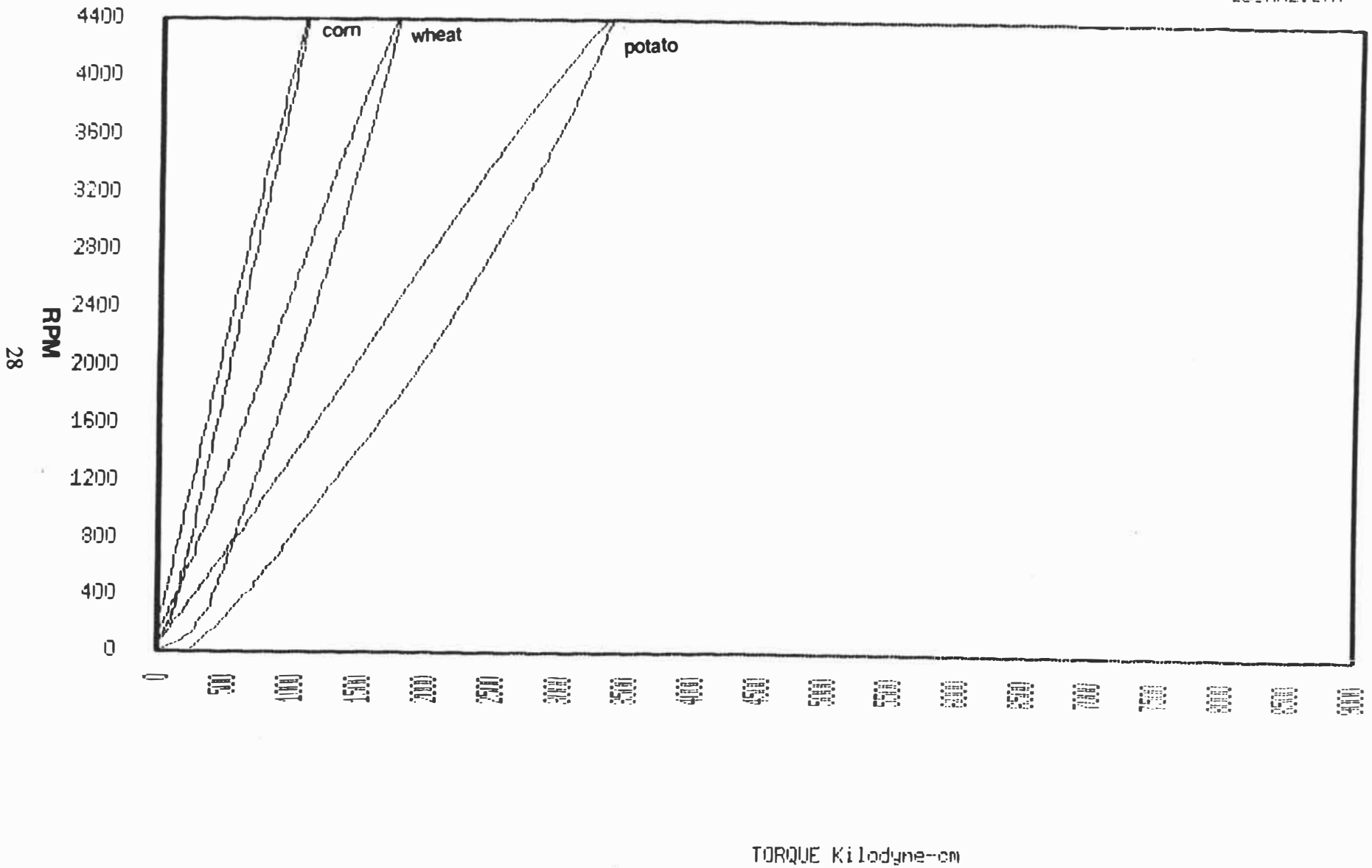


Figure 8 - RPM vs. Torque on Starch/Clay Coatings

BCORN2.DAT
BWHEAT2.DAT
BPOT2.DAT

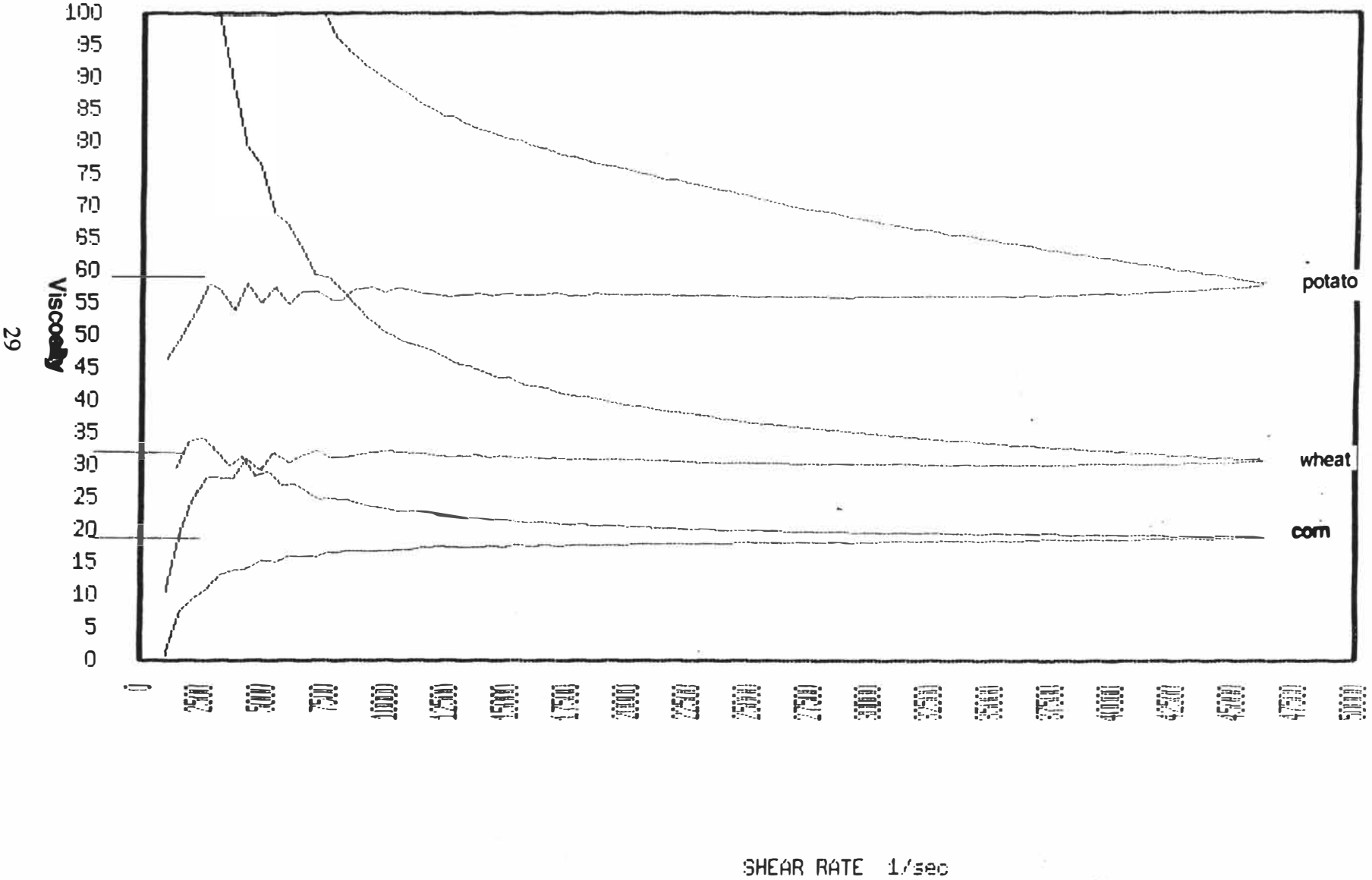


Figure 9 - Viscosity vs. Shear Rate on Starch/Clay Coatings

Addition of latex to the starch, the latex reduced the thixotropic properties of the coating and gave a lower viscosity (fig.. 10 and 11). These curves still followed the pattern as the previous curves, with potato giving the highest viscosity and thixotropy value followed by wheat and corn.

The data of figures 6 through 11 are summarized to show the relationships of thixotropy (table 3). Hercules viscosity (table 4), in the thixotropic breakdown of the fluids.

Tables 3 through 5 summarize the data taken from the rheograms of figures 6 through 11.

The Hercules High Shear Viscosity of wheat starch was 1.25 times greater than corn, while potato starch was 2.2 times greater than corn starch. The coatings showed similar ranking of starch Hi-Shear viscosity, with potato starch giving about 3 times higher viscosity than corn starch. The starch-latex clay coatings had 1.5 to 2 times lower viscosity than the starch-clay coatings.

EPOTS.DAT
EMHAETS.DAT
ECORNS.DAT

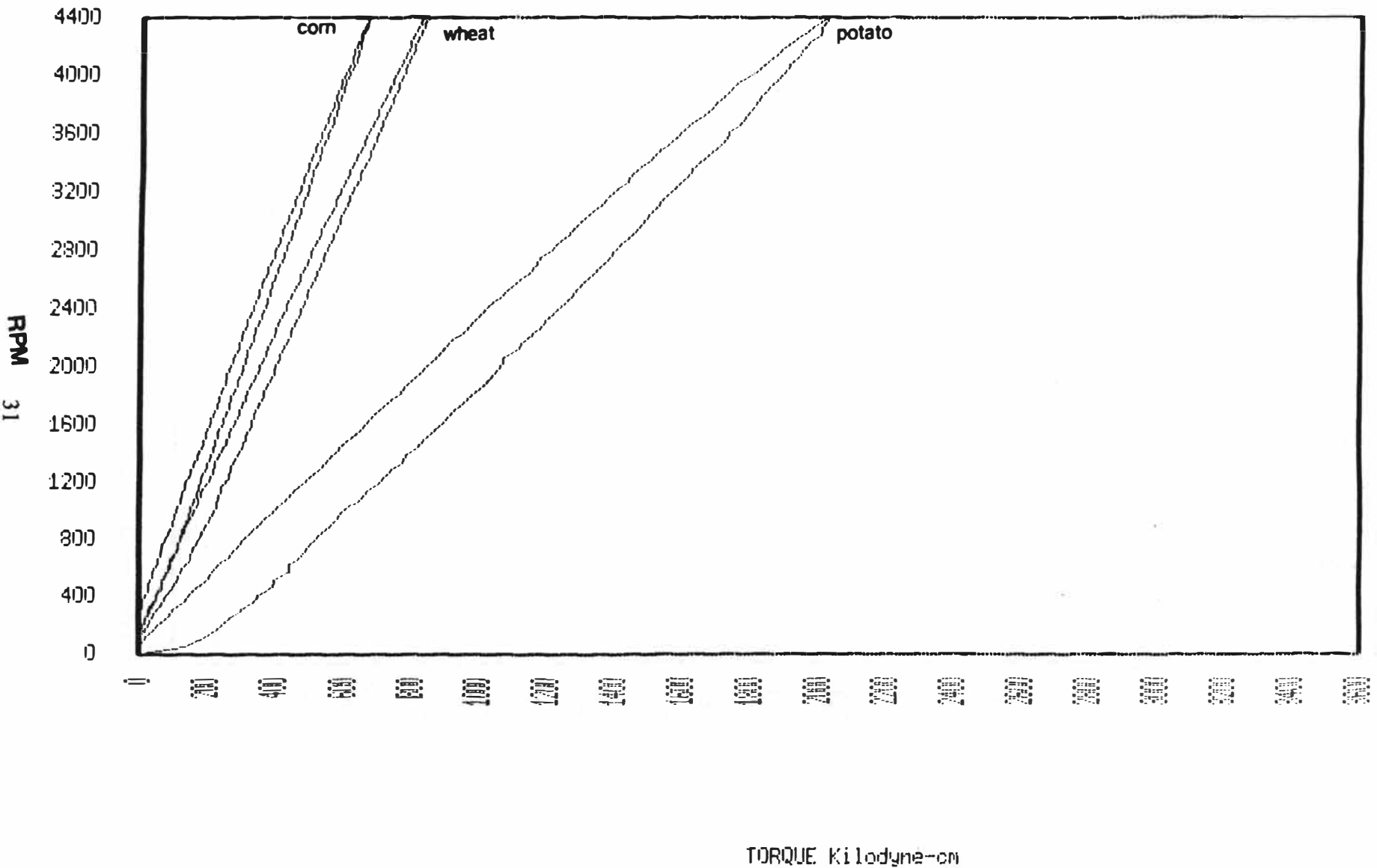


Figure 10 - RPM vs. Torque on 50/50 Starch/Latex Coatings

Table 3

The Coefficient of Thixotropic Breakdown

(All Calculations are 10^{-6} (Appendix A))

	Starch	Starch Coatings	50/50	Starch/Latex
Corn	7.77	60.0		57.2
Wheat	8.82	65.7		30.6
Potato	9.8	105.0		31.8

Table 4

Hercules Hi-Shear Viscosity (4400 sec-1)

	Starch	Starch Coatings	50/50	Starch/Latex
Corn	12	18		12
Wheat	15	32		15
Potato	26	59		36

Table 5

Brookfield Viscosity (cP)

	Starch	Starch Coatings	50/50	Starch/Latex
Corn	325	328		331
Wheat	320	321		335
Potato	315	320		314

RECOMMENDATIONS FOR FUTURE WORK

1. Perform the same experiment using a different conversion method such as thermal-chemical conversion.
2. Perform the same experiment using different starches (tapioca, high-amylose corn, banana, and rice).
3. Perform the same experiment within the same parameters using different viscosity measuring instrument.

Conclusions

Corn Starch

1. Corn starch had the lowest viscosity values (20% lower than wheat and 67% lower than potato) for all the starches tested.
2. Corn starch had the lowest thixotropic breakdown (12% lower wheat and 20.7% lower than potato) for all the starches tested.

Potato Starch

1. Potato starch gave the higher shear viscosity values (20% higher than corn and 50% higher than wheat) of all the starches tested.
2. Potato starch gave the highest thixotropic breakdown (20% higher than corn and 10% higher than wheat) of all the starches tested.

Wheat Starch

1. Wheat starch gave viscosity values between potato and corn starch.
2. The values for thixotropic breakdown in wheat starch was between the potato and corn starch.

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APPENDIX A

Sample formulas for determining thixotropic Breakdown

(For the up flowcurve)

U1 and **U2** = " plastic Viscosities" at different speeds 2200 and 4400 rpms.

T1 and **T2** = Torque (kilodyne - cm)

s = s-factor for the E bob

RPM1 and **RPM2** = 2200 and 4400 respectively

Plastic viscosities are calculated from two points on the up flowcurve of a rhegram corresponding to a set of rotational speeds and torques, (**RPM1,T1**) and (**RPM2, T2**).

$$\mathbf{U1} = \frac{9.55 * \mathbf{T1} \mathbf{s}}{\mathbf{RPM1} \mathbf{b}} \quad \text{and} \quad \mathbf{U2} = \frac{9.55 * \mathbf{T2} \mathbf{s}}{\mathbf{RPM2} \mathbf{b}}$$

where parameter **b** represents the slope and it is equal to:

$$\mathbf{b} = \frac{\ln \frac{\mathbf{RPM1}}{\mathbf{RPM2}}}{\ln \frac{\mathbf{T1}}{\mathbf{T2}}}$$

(for the down flow curve)

U'1 and **U'2** = "plastic viscosities" at different speeds 2200 and 4400 rpms

T'1 and **T'2** = torque (kilodyne-cm)

s = s-factor for the E bob

RPM'1 AND **RPM'2** = 2200 and 4400 respectively

where:

$$\mathbf{U'1} = \frac{9.55 * \mathbf{T'1} \mathbf{s}}{\mathbf{RPM'1} \mathbf{b'}} \quad \text{and} \quad \mathbf{U'2} = \frac{9.55 * \mathbf{T'2} \mathbf{s}}{\mathbf{RPM'2} \mathbf{b'}}$$

and

$$\mathbf{b'} = \frac{\ln \frac{\mathbf{RPM'1}}{\mathbf{RPM'2}}}{\frac{\mathbf{T'1}}{\mathbf{T'2}}}$$

The coefficient of thixotropic breakdown is then defined as:

$$\mathbf{M} = (\mathbf{b}-1)[2(\mathbf{U2}-\mathbf{U1})] - (\mathbf{b}') [2 (\mathbf{U'2}-\mathbf{U'1})]$$